IS 4523 (1989): Acetoacetanilide [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

(Reaffirmed 2000)

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(पहला पुनरीक्षण)

Indian Standard

ACETOACETANILIDE—SPECIFICATION

(First Revision)

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BUREAU OF INDIAN STANDARDS
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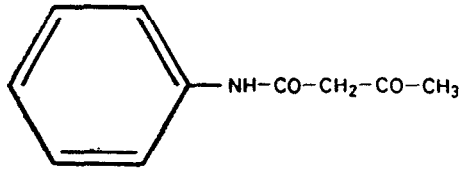
Group 2

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 13 September 1989, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Acetoacetanilide ($C_{10}H_{11}O_2N$) is an important dye intermediate used in the manufacture of yellow organic pigments. It has the following structural formula:



Acetoacetanilide
(Molecular mass 177·2)
CAS Registry Number (102-01-2)

This standard was first issued in 1968. The Committee responsible for its preparation decided to revise it in order to introduce the chromatographic method for estimation of aniline. The requirement of melting point has also been modified.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ACETOACETANILIDE — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for acetoacetanilide.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
1070 : 1977	Specification for water for general laboratory use (*second revision*)
2552 : 1979	Specification for steel drums (galvanized and ungalvanized) (*second revision*)
5299 : 1969	Methods of sampling and tests for dye intermediates

3 REQUIREMENTS

3.1 Description

The material shall be in the form of white crystals.

3.2 The material shall also comply with the requirements given in Table 1.

Table 1 Requirements for Acetoacetanilide

(*Clauses* 3.2, 5.3.1, 5.3.2, 6.1 and *Annex* A)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Annex A
(1)	(2)	(3)	(4)
i)	Melting range	83 to 85°C	A-1
ii)	Matter insoluble in sodium hydroxide solution, percent by mass, *Max*	0·2	A-2
iii)	Aniline, percent by mass, *Max*	0·2	A-3
iv)	Assay, percent by mass, *Min*	98	A-4

4 PACKING AND MARKING

4.1 Packing

Unless otherwise agreed, the material shall be suitably packed either in wooden barrels, multi-walled paper sacks, or in suitable drums (*see* IS 2552 : 1979).

4.2 Marking

The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer, and his recognized trade-mark, if any;

c) Batch number; and

d) Net, gross and tare mass.

5 SAMPLING

5.1 The representative samples of the material shall be drawn as prescribed in **3** of IS 5299 : 1969.

5.2 Number of Tests

5.2.1 Assay and the test for the determination of melting range shall be conducted on each of the individual samples separately.

5.2.2 Test for the determination of matter insoluble in sodium hydroxide solution and aniline content shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of assay and melting range, if each of the individual test results as obtained in **5.2.1** satisfies the corresponding requirements given in Table 1.

5.3.2 *For Composite Samples*

The lot shall be declared as conforming to the requirements of matter insoluble in sodium hydroxide solution and aniline content, if the test results as obtained in **5.2.2** satisfies corresponding requirement given in Table 1.

6 TEST METHODS

6.1 Tests shall be carried out as prescribed in the appropriate clauses of Annex A indicated in col 4 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Table* 1 and *Clause* 6.1)

METHODS OF TEST FOR ACETOACETANILIDE

A-1 DETERMINATION OF MELTING RANGE

A-1.1 Determine the melting range of the material as given in **8** of IS 5299 : 1969.

A-2 MATTER INSOLUBLE IN SODIUM HYDROXIDE SOLUTION

A-2.1 Reagent

A-2.1.1 *Sodium Hydroxide Solution,* 1 N.

A-2.2 Procedure

Take 10 ml of the soidum hydroxide solution in a 100-ml volumetric flask add 1 g of the material. Shake well and allow the solution to stand for 5 minutes. Make up the volume with water. Filter through a sintered crucible of porosity G4, wash thoroughly dry, weigh and calculate the percent matter insoluble in alkali.

A-2.3 Calculation

$$\text{Matter insoluble in sodium hydroxide solution, percent, by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of material taken for the test.

A-3 DETERMINATION OF ANILINE

Thin layer chromatographic method is employed for estimation of aniline.

A-3.1 Apparatus

A-3.1.1 *Thin Layer Chromatographic Plate*

Glass plate of size 10 cm × 20 cm, coated uniformly with silica gel G of 250 micron thickness and activated at 110°C for 30 minutes.

A-3.1.2 *Micropipette* — 10 µl capacity.

A-3.1.3 *Developing Chamber* — suitable rectangular glass jar which contains the developer and which is closed well with the lid for saturation.

A-3.2 Reagents

A-3.2.1 *Methyl Alcohol*

A-3.2.2 *Fast Blue B Salt* (*Tetrazotised-o-dianisidine zinc double salt*), 1·0 percent solution in water.

A-3.2.3 *American Base* [*N-(1 Naphthyl) ethylene-diamine dihydrochloride*], 0·1 percent solution in methanol.

A-3.2.4 *Eluent,* A mixture of butyl acetate and acetic acid (99 : 1).

A-3.3 Procedure

A-3.3.1 *Preparation of Sample Solution*

Dissolve 0·2 g (on 100 percent basis) of sample in 5 ml methanol (4·0 percent solution).

A-3.3.2 *Preparation of Standard Solutions*

i) Prepare (standard) solution as above ii) Prepare 0·004 percent solution in aniline in methanol.

A-3.3.3 Spot 10 µl of standard and sample solution on the first plate. Spot 10 µl of standard and sample solution and aniline solution on the second plate. Allow to dry. Then place the plates in the solvent of the developing chamber in a vertical manner and close the chamber. Allow to run for 13 cm. Take out the plates and dry the solvent completely. Spray the first plate with Fast Blue B salt solution (**A-3.2.2**). Compare the intensity of the spot visually with those of the known standard (yellow single spot).

Diazotise the second plate by putting it into a chamber containing nitrous fumes for 1 minute and then spray the plate with American base solution (**A-3.2.3**). Compare the intensity of the spot with the spot of known concentration of aniline for both standard as well as sample. The maximum allowable limit for aniline in standard as well as sample is 0·1 percent.

A-4 ASSAY

A-4.0 Outline of the Method

A standard diazonium solution is prepared by reacting *p*-chloroaniline with standard sodium nitrite solution. The sample of the acetoacetanilide is then titrated with standard diazonium solution.

A-4.1 Reagents

A-4.1.1 *Hydrochloric Acid,* 30 percent (*m/v*).

A-4.1.2 *Standard Sodium Nitrite Solution,* 1 N, freshly standardized.

A-4.1.3 *p-Chloroaniline*, 99 percent pure.

A-4.1.4 *Sodium Hydroxide Solution*, 40 percent (*m/v*).

A-4.1.5 *Dilute Acetic Acid*, 25 percent (*m/v*).

A-4.1.6 *Pyridine* (*water white grade*), 99°/160°C.

A-4.1.7 *H-Acid Indicator Solution*

Dissolve 0·5 g H-acid in 100 ml water containing 1 g of soda ash.

A-4.1.8 *Tetrazodianisidine Solution*

Take 2 g of dianisidine base in a beaker and dissolve in 7 ml of hydrochloric acid (heat, if necessary up to 50°C). Cool with ice to about 0°C and add immediately 12 ml of sodium nitride solution and make up the volume to 100 ml with ice cold water. Shake mildly. Test for excess sodium nitrite with starch-iodide test paper. Store this solution in an amber coloured bottle in a cool place.

A-4.1.9 *Starch-Iodide Test Papers*

A-4.2 Procedure

A-4.2.1 *Preparation of Standard Diazonium Solution of p-Chloroaniline*

Take about 4 g of pure *p*-chloroaniline in a 250-ml glass beaker containing 10 ml hydrochloric acid and 150 ml water, heat up to 60°C to dissolve completely. Cool externally to 0 to 5°C with chopped ice, titrate with standard sodium nitrite solution with gentle stirring. Test the solution for excess of sodium nitrite by spotting on starch-iodide test paper. The end-point is reached when an immediate faint blue coloured ring appears on starch-iodide test paper which may be obtained repeatedly during a period of 10 minutes without further addition of sodium nitrite solution. Allow to stand for 30 minutes. Filter to remove insoluble matter and then make up the filtrate to 250 ml in a volumetric flask. Store the standard diazonium solution in an ice bath in the dark.

A-4.2.2 Weigh accurately about 0·660 g of acetoacetanilide test sample and dissolve with 1 ml of sodium hydroxide solution in 25 ml of water, stir this solution and add drop-wise dilute acetic acid till the *p*H is 7 which may be judged by using *p*H paper. Dissolve under good stirring precipitated acetoacetanilide with 25 ml pyridine and few drops of methanol to get a clear solution. While stirring mechanically add the standard diazonium solution (*see* **A-4.2.1**) from a burette equipped with water jacket through which water is circulating at about 10°C. The burette should be of amber glass to minimize any decomposition of the diazonium salt by light. Titrate as rapidly as spot test permits. To test for excess acetoacetanilide place a few drops of titration mixture on a filter paper. About one centimetre away from the edge of the liquid mark, place a few drops of tetrazodianisidine solution. Where the two liquid portions meet on the filter paper, a brown colour will develop if excess of acetoacetanilide is present. Similarly try with H-acid indicator to test the excess of diazonium salt. If a pink colour develops at the interjunction, excess of diazonium solution is indicated.

A-4.2.3 Initially add diazonium solution in 1 to 2 ml portions, testing titration mixture after each addition for excess of acetoacetanilide and diazonium salt. As the end-point approaches, diazonium standard solution should be added in portions of 0·2 ml. The end-point is reached when no reddish brown colour is given with tetrazodianisidine solution and no pink colour or very faint pink colour is given with H-acid indicator. Note the volume of diazonium solution required for titration.

A-4.3 Calculation

$$\text{Assay, percent by mass (calculated on molecular mass 177·2)} = \frac{V \times 17{\cdot}72 \times N}{M}$$

where

V = volume in ml of standard diazonium solution required,

N = normality of the standard diazonium solution, and

M = mass in g of the sample taken for the test.

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